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First Quarterly Report

for

CHARACTERIZATION OF NICKEL-CADMIUM ELECTRODES

1 July, 1963 - 1 October, 1963

Contract No. NAS5 - 3477

Prepared by

General Electric Company

Advanced Technology Laboratories
Schenectady, New York

for

Goddard Space Flight Center
Greenbelt, Maryland

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Summary

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The objective of this contract is to develop a method of analysis and characterization of the electrodes used in nickel-cadmium sealed cells. It is based primarily on a comparison of detailed polarization measurements of single electrodes before and after periodic operation in selected modes of cyclic testing of cells at three temperature levels 0°C, 25°C and 50°C. A correlation of this data should provide a basis for specifying improved cells for space applications as well as comparing cells from various manufacturers.

The work during the first quarter was largely of a preparative nature. It involved the design and construction of the extensive control and testing equipment required for conducting the characterization and cyclic tests; design and construction of test cell holders; and the preparation of electrodes for the test program.

AUTHOR

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1.0 Introduction

This report covers the work done during the first quarter of an 18 month program to develop a method for the analysis and characterization of the electrodes used in nickel-cadmium spacecraft batteries.

The goal of the program is to develop a correlation between detailed characterization data obtained on single electrodes and the behavior of these electrodes in cells in various modes of cyclic operation. Such a correlation will provide a basis for specifying improved cells for space application and comparing cells from various manufacturers.

The characterization will be made by analysis of data taken in single electrode experiments based on the use of continuous recording of charge-discharge curves under various testing regimes. The effects of environmental and operating factors on electrode behavior, plus those attributed to manufacturing and assembly techniques will be measured and evaluated. The characterization information will include: 1) polarization of each electrode under various conditions, 2) complete charge and discharge curves showing electrode capacity, impurity levels, onset of gassing, graphitic and antipolar capacity, and reproducibility of cell operation, and 3) the onset of changes in capacity under various operating conditions.

Periodically test cells will be removed from tests and the individual electrodes will be recharacterized and examined for changes in physical properties and comparisons made to the original characterization data.

Work accomplished during the first quarter included the design, procurement and construction of electrical test equipment to conduct the characterization tests on single electrodes, as well as equipment for the cyclic testing of cells; and the construction of test cell holders for electrode characterization and cyclic tests.

During the second quarter, the equipment will be completed and experimental work on characterization and cyclic testing will be initiated. By the end of the 2nd quarter all cyclic tests will be underway, which will allow for approximately 12 months of testing.

2.0 Program Scope

A breakdown of the program into tasks is given in Table I along with a brief description of the tasks. The first three tasks are of a preparative nature. Tasks IV through VII constitute the experimental portion of the program.

The test program is divided into two parts: one, the characterization testing of plates; and the other, the cycling of cells made from the characterized plates. After a period of cycling, the plates will be retested, and returned to cycling. A correlation between cycling variable and changes in plate characterization will be made. This correlation will aid in determining what characterization tests are most useful in the evaluation of nickel-cadmium cells.

In the program, the following general procedures will be followed:

1. All plates in the program will be SAFT type VO, prepared as for space cells.

2. Tests will be grouped to run concurrently at 0^{o}, 25^{o} and 50^{o} C.

3. Failure analysis on cycled cells will be made, using visual, mechanical, chemical, and electrochemical procedures.

As the program proceeds, new characterization tests suggested by the results will be devised and plates tested.

TABLE I
PROGRAM TASKS
NAS5 - 3477

	TASK		MEASUREMENTS
I	Control and Recording Equipment Design and fabrication of test equipment.		None
II	Test Cell Assemblies Design and fabrication of test cells.		None
III	Electrode Preparation Electrochemical cleaning of reference and cell electrodes, inspection and welding of identification tabs.		Capacity check and weight
IV	Characterization Tests - CA and CB	C-A	Determine charge curve
	C-A Constant current charging at C/10, C/5 and C		Determine rate of gassing from electrodes
			Determine rate of O_2 recombination.
	C-B Constant current discharging at C/10, C/5 and C	C-B	Determine discharge curve
V	Shallow Discharge Cycling Tests C-C	C-C	Make periodic capacity determination.
	Constant current cycling to 25-35% range to determine memory effects		Make analysis of physical properties
			Recovery test
VI	Random Discharge Tests R-A	R-A	Periodic charge and discharge curve.
	Random discharges averaging 10%, 25%, 50% and 75% depth of discharge over a 6 day period using Gaussian and rectangular distribution for discharge periods.		Recharacterization tests.
VII	Constant voltage, current limited charging cycling tests R-B charging at C/5 rate and discharge at C/2 rate to 0.9 volts.	R-B	Periodic charge and discharge curve.
			Recharacterization tests.

3.0 Discussion

During this quarter work was initiated on the first three tasks and plans made for implementing the testing program during the second quarter. The status of this work by tasks is reported here.

3.1 Control and Recording Equipment

The basic functions of the equipment for the characterization tests and the cycling tests are all very similar, namely provide for: controlled charge and discharge rates; controlled times and switching for each portion of the cycle; and measuring and recording the potential of cells against a reference electrode or the total cell potential. The basic circuit for these functions is shown in figure 1.

The detailed circuit schematics of the control portion of the equipment for the CA-CB, C-C, R-A and R-B are in appendix I.

Approximately 75 percent of the work to fabricate these units was accomplished during the quarter. All components for completion are on hand except for one item. This is a light actuated programmer for use in controlling the random discharge cycles for the R-A tests. This item is scheduled for delivery by the middle of November.

Each of the units will have sufficient channel capacity to allow for concurrent testing at the three temperature levels. This equipment has been purchased and is being constructed on company funds.

3.2 Test Cell Assemblies

The test cells for the majority of the characterization tests and cyclic testing will require two basic types of construction: a flooded cell construction for the majority of the characterization tests and a starved cell construction for all of the cycle tests.

The basic features of the flooded cell construction are shown in figure 2.

The starved cell construction is achieved by eliminating the grooves from the interior of the cell holder. The reference electrode in this case is a full size plate other than the cut down version used in the flooded cell construction. An exploded view drawing of the construction is shown in figure 3. The packing pressure of the plates will be controlled by installing the cell pack into the partially completed holder, inserting the face plate piece and loading the stack with a fixed weight. The face plate is then sealed while under load.

The parts for these cells have been machined and are ready for assembly as soon as the plates have been prepared for testing.

Special cell holders will be designed for the tests to determine the gassing and recombination rates characteristics of the electrodes. For the gassing tests a flooded cell construction is contemplated employing either a finely porous teflon or ion exchange membrane to minimize diffusion of oxygen to cadmium where it would recombine. The recombination cell holder will be of the starved construction type.

In each case provisions will be incorporated for monitoring the pressure rise in a calibrated fixed volume reservoir. These cell assemblies will be fabricated during the second quarter.

3.3 Electrode Preparation

The plates to be used in the program are standard Saft VO types measuring approximately 1-3/4" x 3" and have a nominal capacity of 1.0 amp-hr per two plate cell. Approximately 550 plates will be required for the test program. These are on hand and work to prepare these for the test program was initiated during this quarter and will be completed early in the second quarter.

A special procedure for cleaning these plates has been adopted. The electrochemical cleaning process consists of a number of polarization cycles, washing, drying and inspection for gross visual defects. The details of the processing are given in Table II.

This cleaning is done to insure complete activation of active material; supplement the removal of any contaminating ions; establish that there is a proper balance of positive and negative capacity in the selected plates; bring the plates to the proper state of discharge.

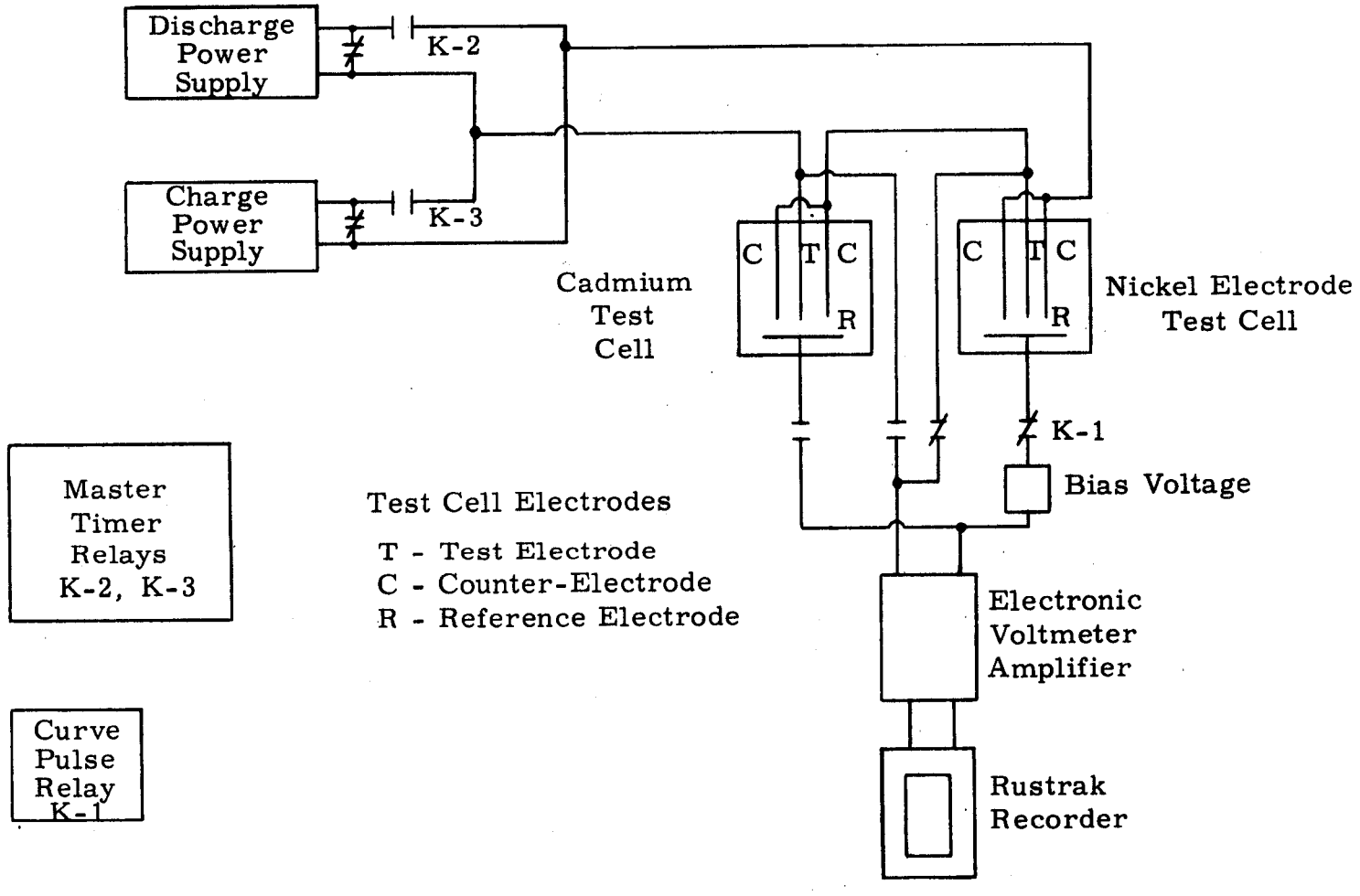
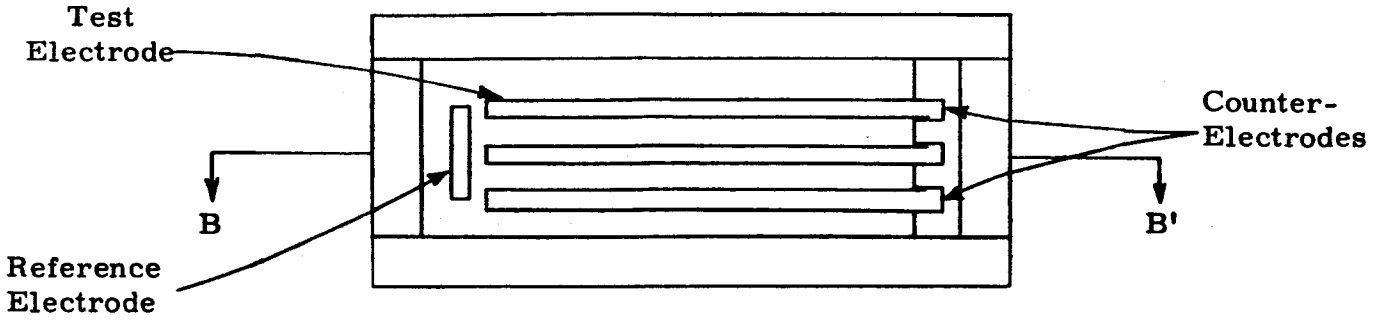
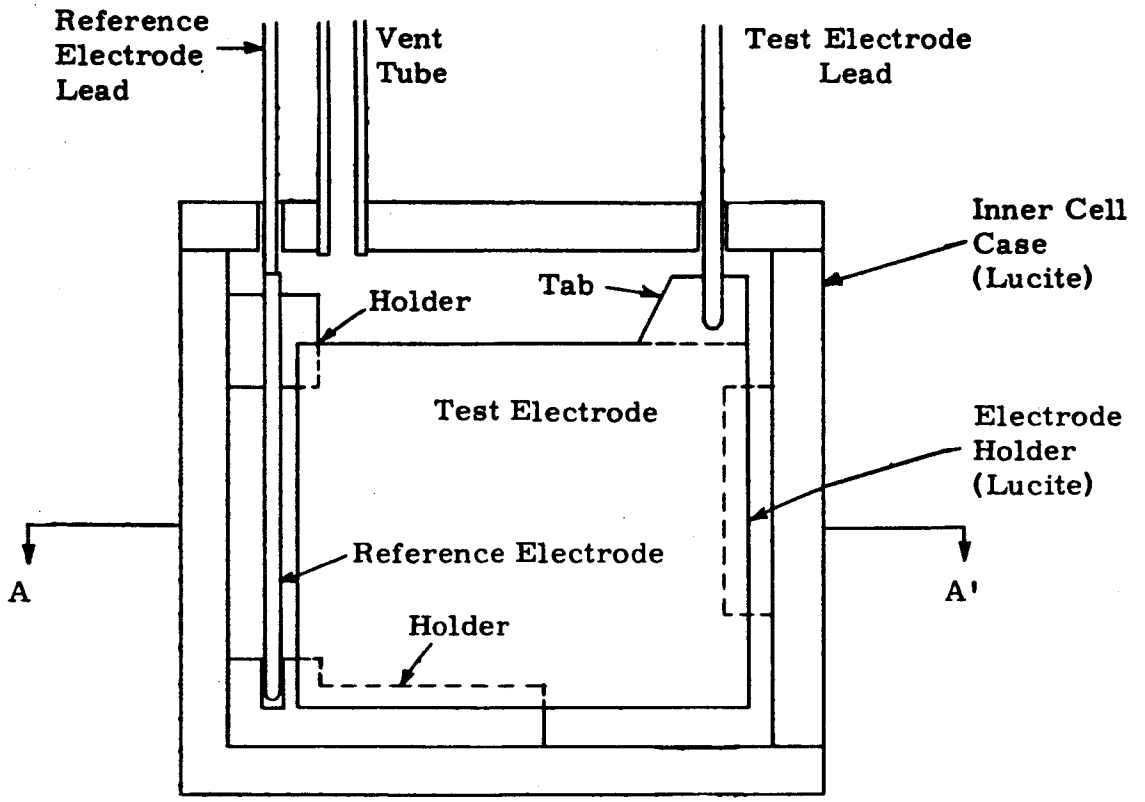


Fig. 1. Basic Cycling Test Circuit.



A-A' Section



B-B' Section

Fig. 2 Flooded Type Test Cell.

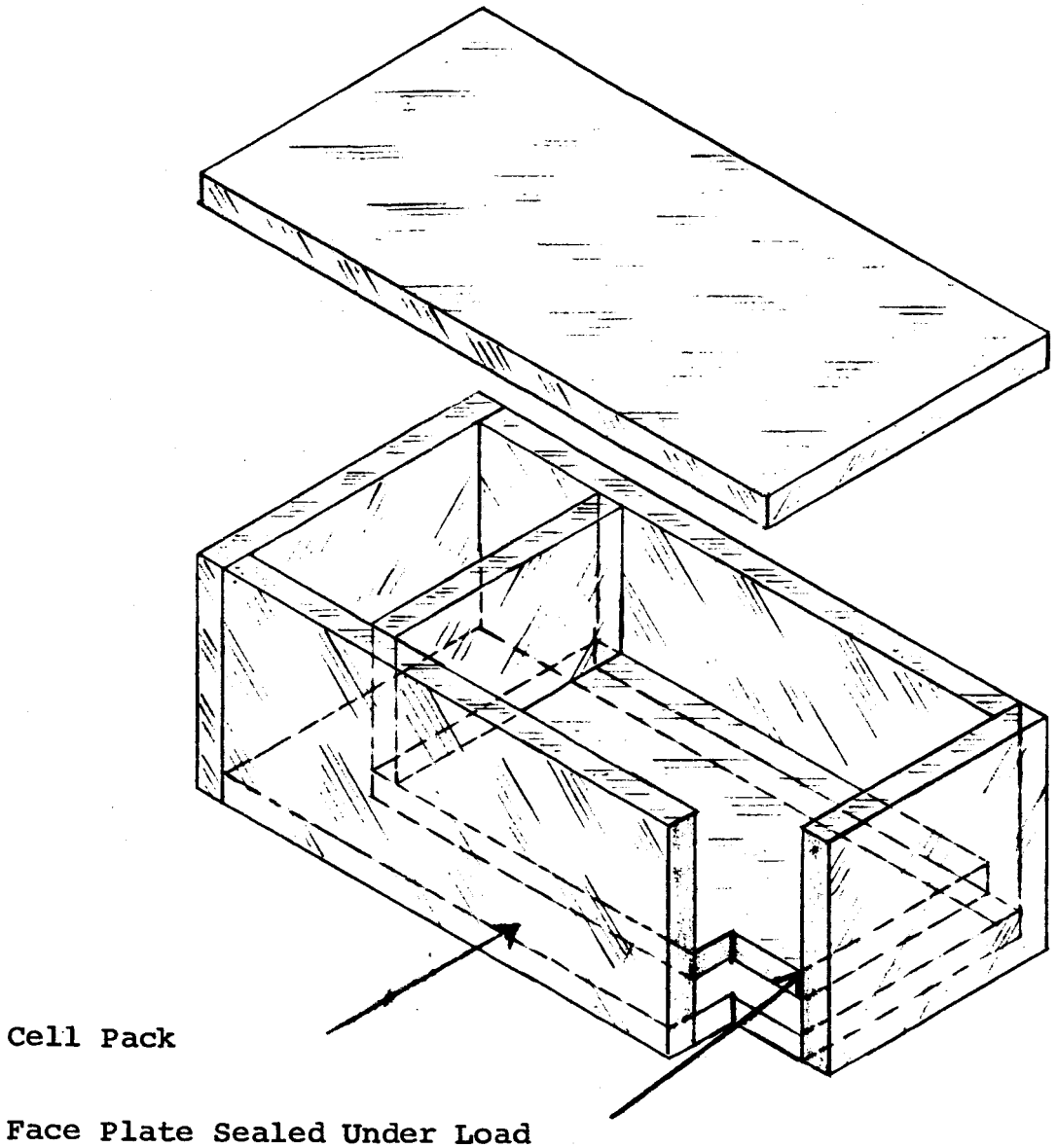


Fig. 3 Starved Type Test Cell.

Table II

ELECTRODE CLEANING PROCESS

1. The cells are immersed in 25% KOH in separate compartments and allowed to soak for 16 hours. If the cells are degassed under vacuum, the soak time may be reduced to two hours.

2. The cells are connected in series and discharged through inversion at C/8 amps. for 4 to 6 hours. C is the nominal capacity in ampere hours. The individual cell voltages should be greater than 1.50 V at the end of the discharge. If the value is less than this, the inversion is not complete and the discharge must be continued until a value greater than 1.50 V is obtained.

3. The polarity is reversed and the cell charged at C/8 amps for 16 to 20 hours. The voltage must reach 1.5 V.

4. The cells are discharged at approximately C/2.5 to zero. The cells may be directly shorted with a jumper when the discharge voltage is less than 0.6 V.

5. The individual resistors (or jumpers) are removed and the cells in series are discharged through inversion at C/12 for 7 hours.

6. Charged as in 4.

7. Discharged as in 5. The capacity obtained must be at least 125% of C.

8. Individual cells are shorted for a minimum of 1 hour. .

9. The cells in series are discharged through inversion at C/12 for a maximum of 6 hours.

   When a cell reaches minus 1.2 V it shall be electrically removed from the discharge circuit. The discharge may then continue on the balance of the cells.

   Note: The individual cell voltage after 3 hours of inversion must be between minus 0.1 and minus 1.0 V. If this value is negative in excess of minus 1.0 V, the cell does not contain sufficient excess negative capacity.

10. The separator is removed and the plates washed in deionized water until the pH of the final wash water is less than 10.5.

11. The plates are dried in a forced air oven at $100^{o}C \pm 10^{o}$ for 2 hours.

12. The plates are removed from the temporary assembly and inspected for defects. Any plate showing evidence of blistering, disintegration or severe pitting is rejected. Any smut or loose active material is removed from the surface by brushing with a dry nylon brush or by wiping with a clean, dry cloth or paper towel.

13. The plates are stored in sealed plastic containers until used.

3.4 Test Program Tasks IV through VII

No experimental work was done on these tasks during the quarter. Some of the salient parts of the testing program procedures and data to be obtained and correlated are described in this section:

The number of cell assemblies and plates per cell to be used in the program are given in Table III.

3.4.1 CA-CB Tests

As has been indicated previously the key characterization data to be obtained in the CA-CB tests is a complete potential versus time curve for each electrode for a small number of charge-discharge cycles (5-10) prior to assembling them into the test cells for the cyclic tests. These tests will be conducted in cells employing a well aged nickel oxide reference electrode and counter electrodes as shown in figure 2. The reference electrode is (about 10%) fully charged and then discharged and aged for several weeks. The potential of the electrode remains stable over a period of months if currents are not drawn from it. It can be renewed in the test cell if necessary.

In use, a pair of test cells, one with a cadmium test electrode, the other with a nickel-oxide test electrode are run thru the cycling together. A master timer is used to turn on the charge and discharge power supplies thru appropriate relays. A curve pulser relay is used to cycle the input to the amplifier between the nickel-reference pair and the cadmium-reference pair at intervals of 1-2 min. The recording gives two curves, one for each electrode. The bias voltage is set to cancel out the reference voltage so that the difference between the curves is the cell voltage that would be observed if the pair of test electrodes were in a cell. The electronic voltmeter is used to avoid current drain on the reference electrode. Alternate recording of the two curves is possible since the Rustak recorder is a printing type rather than a continuous curve tracer.

Figure 4 shows a typical set of curves for the potential of cadmium and nickel electrodes during a test cycle. This set of curves was taken using constant charging and discharging current, a nickel electrode reference, and a set of plates from a General Electric type VO-4 nickel-cadmium cell. The detail of information shown is apparent.

All electrodes will be characterized in this manner and compared with similar curves obtained on the electrodes which will be periodically removed from the cyclic tests C-C, R-A, and R-B. The electrodes will be divided into three equal groups with each group being characterized at one temperature

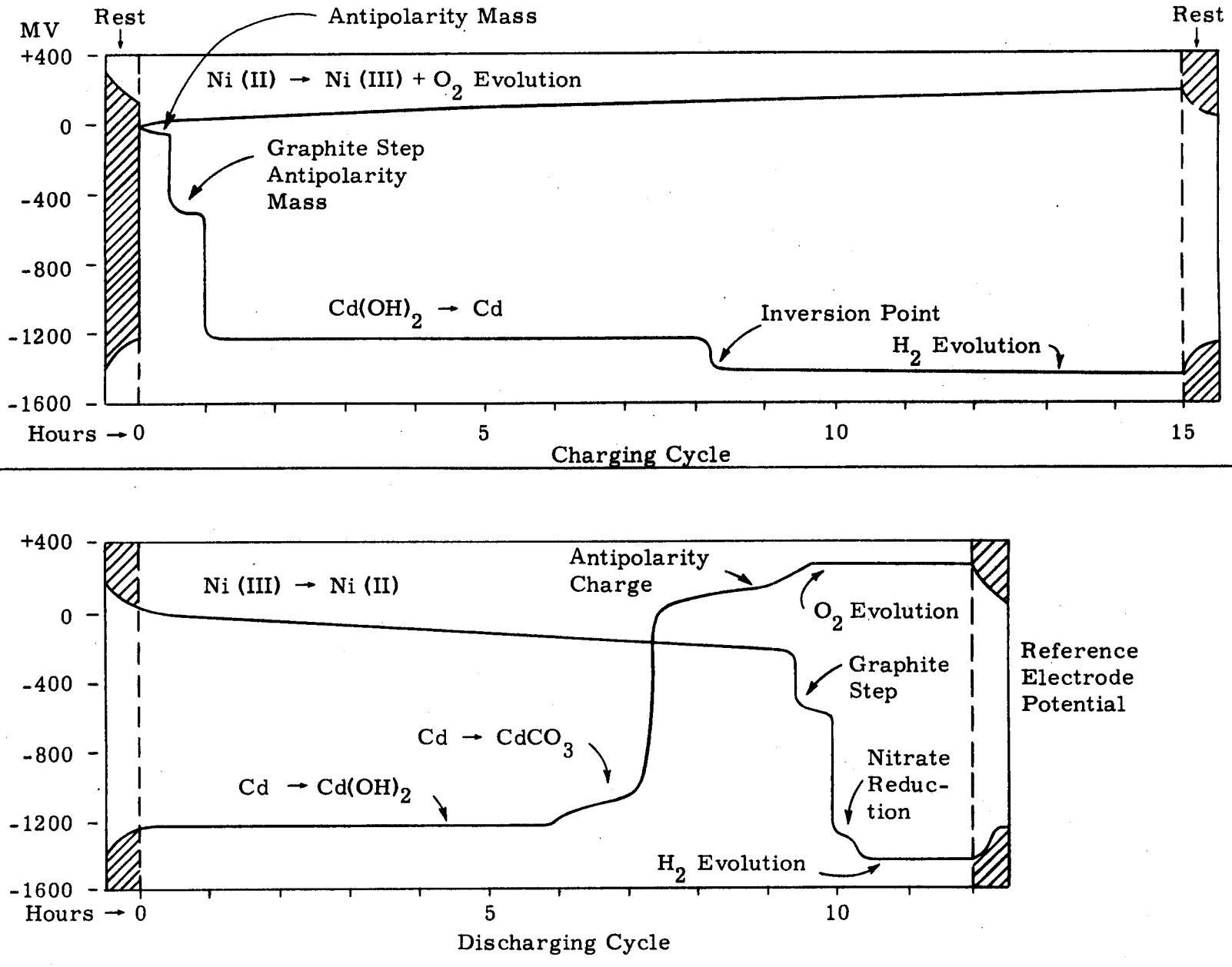


Fig. 4 Typical Test Curves.

level only. The majority of the electrodes will be characterized in the flooded construction type cell holders. A limited number of electrodes will be characterized in the starved type holders to determine if there are any differences between the two conditions of operation.

The initial gassing and recombination characteristics will be established on a limited number of plates as indicated in Table III. The exact number to be tested will be determined after the initial test results are examined for reproducibility. These results will serve as a reference point for comparison with the results of plates which will be periodically removed from the cyclic tests.

3.4.2 C-C Tests

These tests are designed to determine the onset and severity of memory effects.

A total of 9 cells each containing 4 nickel electrodes and 5 cadmium electrodes will be used in these tests; 3 cells per temperature level. These will be cycled at constant current simulating the condition of a 90 minute orbit, 35 minute discharge and 55 minute charge. The depth of discharge will be limited to 25%.

Periodically, a test cell will be removed from the cycling tests, and a pair of electrodes will be individually tested for capacity, using fully charged counter-electrodes, to 0.9 volt at the cycling discharge rate, followed by discharge through a resistor to zero voltage. This will check the capacity of the plate and determine which plate is showing the memory effect and the severity of the effect.

If memory has set-in, a test plate will be removed without discharge and examined by physical methods (x-ray, etc.) to determine what changes if any have occurred. The remaining test plates will be electrochemically tested to determine recovery from memory and characterized. Surviving plates from the tests will be replaced in the cycling to accumulate data.

3.4.3 R-A Tests

These tests are designed to determine the effect of two types of cell operation which cells may encounter in many applications. On any given mission or long period of time the depth of discharge for a given cell in cyclic operation is likely to vary over a broad range of depths of discharge in a random manner ranging from zero to total discharge from one cycle to another. Yet, the average depth of discharge for the period may be a low value such as 10%. The distribution of the depth of discharges over the

period is most likely to be Gaussian or rectangular. The planned series of cycling tests includes the spectrum of 10, 25, 50 and 75% average depths of discharge. A skewed random Gaussian distribution pattern of discharge will be used for all average depths of discharges with the limits of zero percent and 95% for the individual discharge depths. In addition, at the 25% and 50% average depth of discharge level a random rectangular distribution pattern of discharge will be used with the limits of 10% and 80% depth of discharge for the individual cycle discharges.

Periodically the charge-discharge cycle will be recorded for voltage and capacity determinations. At the end of 100 to 250 cycles the test cells will be taken apart and plate recharacterized; and the plates will then be reassembled in cells for further cycling.

3.4.4 R-B Tests

These tests are designed to determine the effect of cyclic operation under constant voltage, current limited charging conditions. Charging will be done at C/5 rate, with the voltage limit set by the temperature of operations, for 8 hours and discharged at the C/2 rate to 0.9 volt. Recharacterization and cycling will be the same as for the R-A tests.

4.0 Program for Next Quarter

The preparative tasks I thru III will be completed. The characterization tests C-A and C-B for electrodes to be used in the cyclic tests will be completed. By the end of the second quarter all of the cyclic test C-C, R-A, and R-B are scheduled to be underway.

Table III

TEST CELL SUMMARY

Test	Temperature 0°C	25°C	50°C	Total Cells	Plates/Cell Ni (Test)	Cd (Test)	Ni (Counter)	Cd (Counter)	Ref.
CA-CB									
Characterization	8	8	8	24	1	1	2	2	1
Gassing	2	2	2	6	1	1	2	2	1
O_2 Recombination	1	1	1	3	2	1	0	0	1
C-C	3	3	3	9	4	4	0	1	1
R-A				18	1	1	0	1	0
10%	1	1	1						
25% G	1	1	1						
25% S	1	1	1						
50% G	1	1	1						
50% S	1	1	1						
75%	1	1	1						
R-B	2	2	2	6	4	4	0	1	

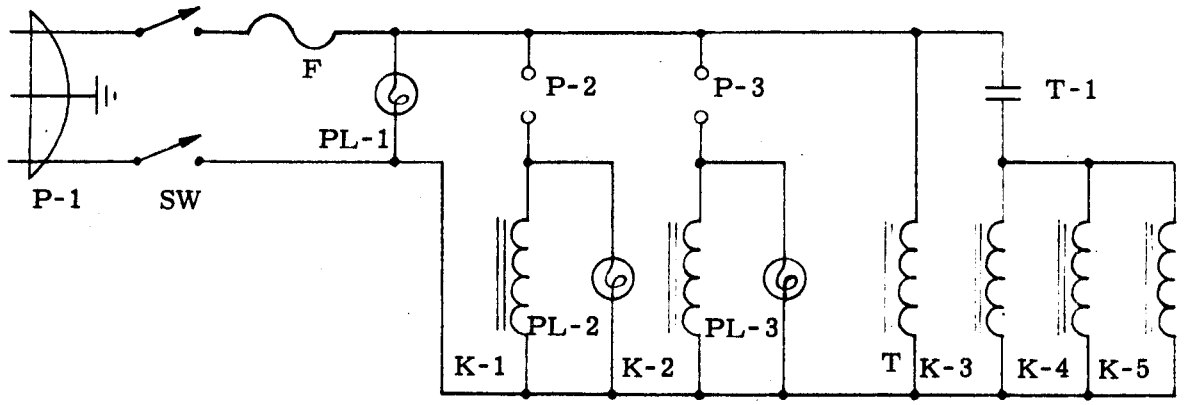
APPENDIX I

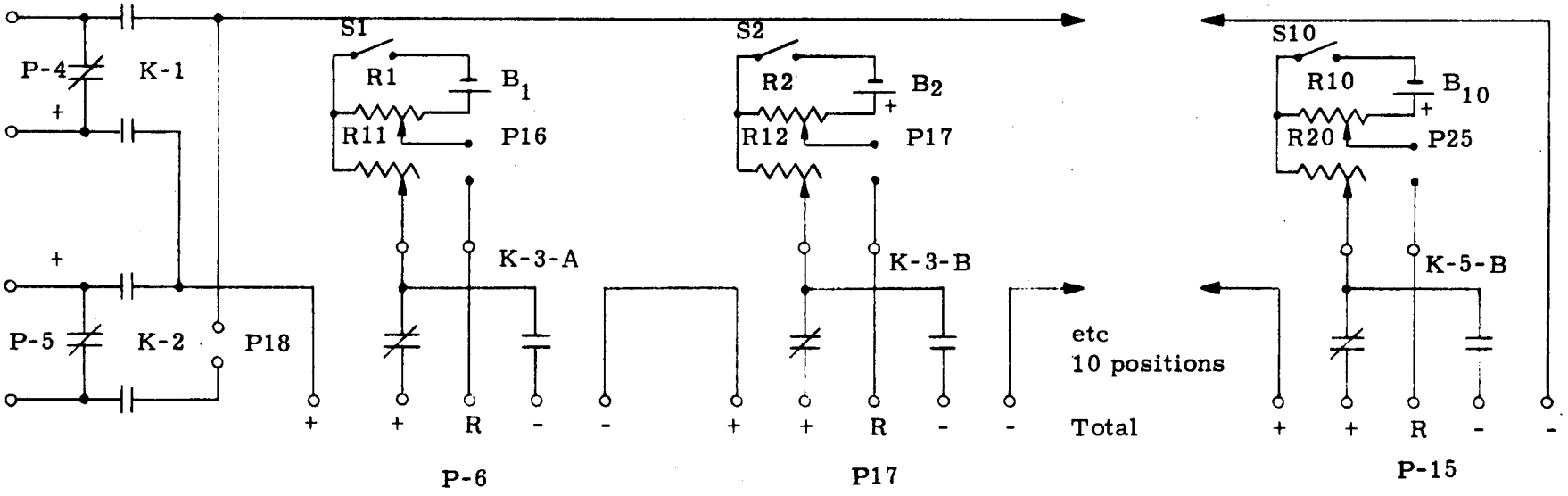
Control Equipment Schematics

Ni-Cd Characterization

CA-CB Cycle Control

Part 1 of 2 Parts





Note: RL changed with variation in load

C/10	500	12.5 Watts	Ohmite 0117
C/2	100	25	Ohmite 0151
C	50	50	Ohmite 0318

PARTS LIST

Part 2 of 2 Parts

P-1 Line Cord, CA-CB Controller

P-2 Control, Charge, Pin Jacks, Yellow

P-3 Control, Discharge, Pin Jacks, Red

P-4 Input Charge, CCS, Terminal

P-5 Input Discharge, CCS Terminal

P-6 thru P-15 Pin Jacks, Cell Input Red +, Green +, White R, Blue -, Black -

P-16 thru P-17 Pin Jacks Recorder, Red +, Black -

P-18 Pin Jacks, G. R. for RL

SW Switch, Toggle SPDT

F Fuse, 3A

PL-1 Pilot, Master, Green

PL-2 Pilot, Charge, Yellow

PL-3 Pilot, Discharge, Red

T Timer, CM, 2 Min

RL Load Resistance (See Drawing)

K-1, K-2 Mercury Relay, 3PST 2NO, 1NC, Ebert EM7, 110 VAC Coil

K-3, K-4 Leach 329-7 4PDT, 110 VAC Coil

K-5 Leach 337 2PDT, 110 VAC Coil

S-1 thru S-10 Switch SPST Toggle "Bias"

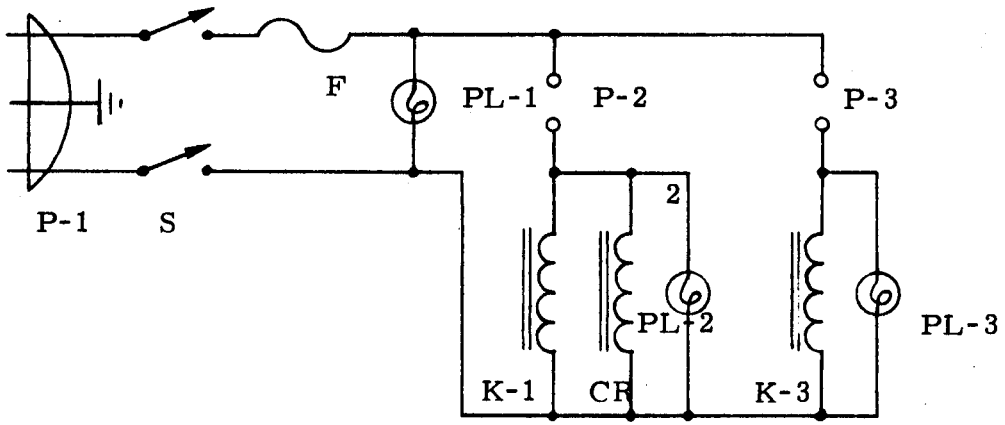
B-1 thru B-10 Battery Mercury O Cell

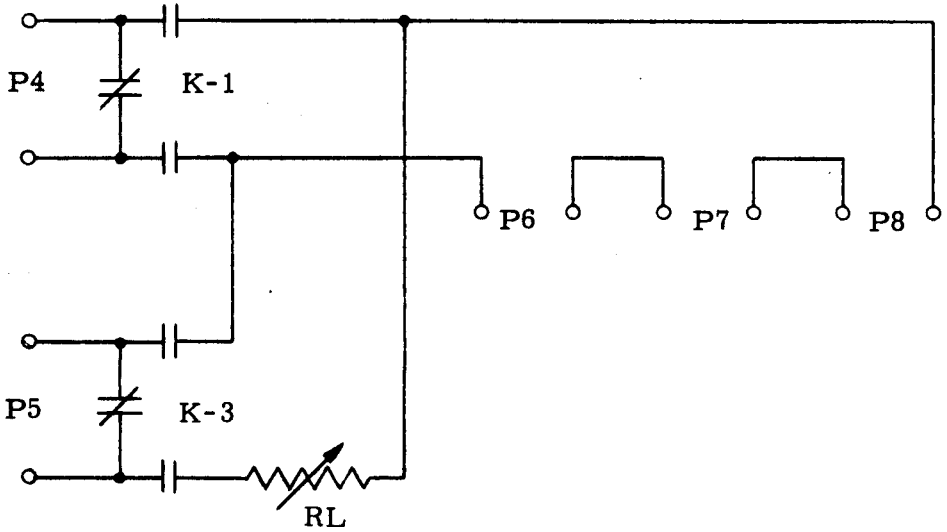
R-1 thru R-10, 50,000 Ω 2 Watt

R-11 thru R-20, 3 Meg

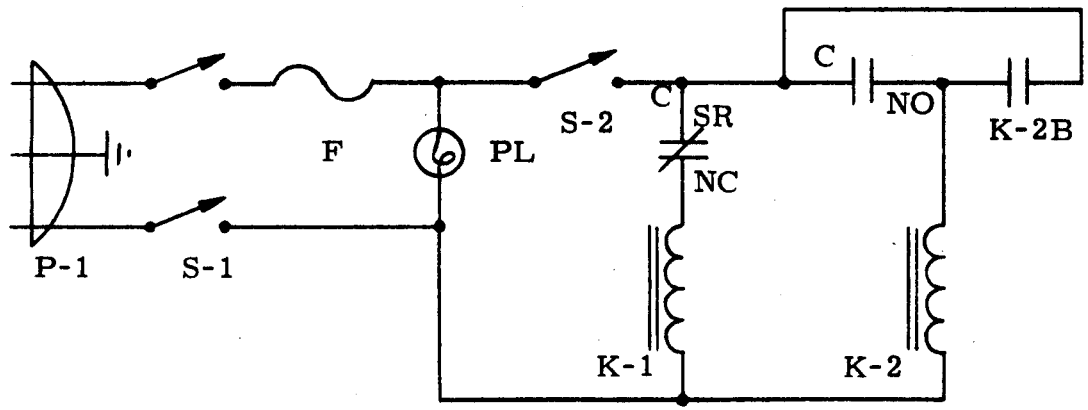
Ni-Cd Characterization

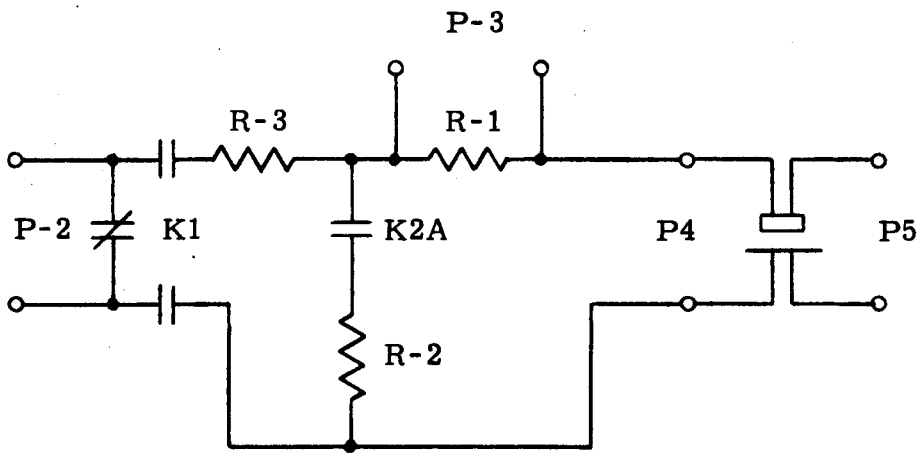
C-C Cycle Test Controller





P-1 Line Cord
P-2 Charge Control Yellow
P-3 Discharge Control Blue
P-4 Charge Input Term Strip, 30 Amps
P-5 Discharge Input Term Strip, 30 Amps
P-6, P-7, P-8 Cell, 30 Amp Term Strip
S Switch Master
F Fuse 5 Amp
PL-1 Master Green
PL-2 Charge Yellow
PL-3 Discharge Red
K-1, K-3 Mercury 3PST 2NO, Ebert INC. EM-7
CR Counter, General Controls CE600BS602
RL 1 Ohm 50 Watts, Ohmite 0309





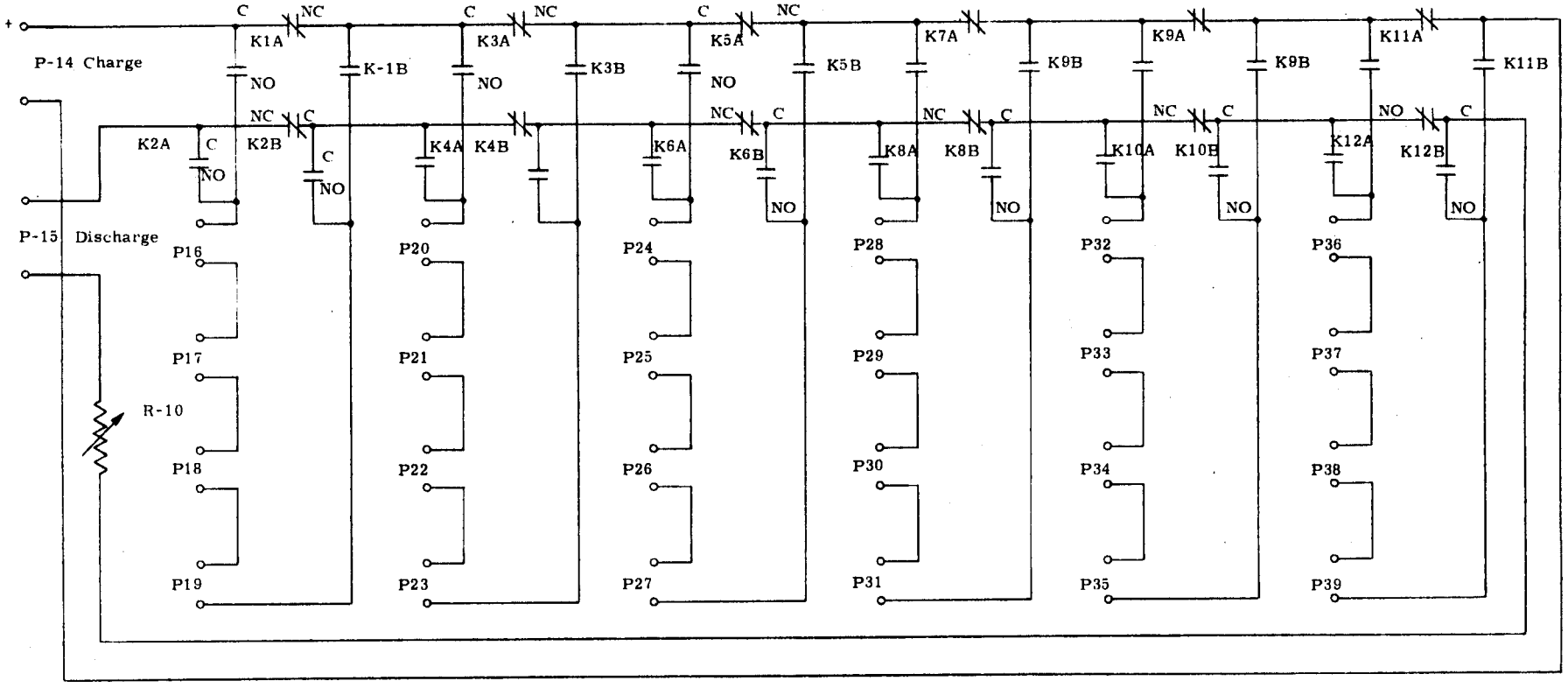
Ni-Cd Characterization

CC Capacity Test Control

P-1	Line Cord
P-2	CC Input Pin Jacks Red + Black -
P-3	Current Recorder Pin Jacks Red + Black -
P-4	Cell Current Terminals
P-5	Voltage Recorder Pin Jacks Red + Black -
S-1	Master Switch
S-2	Start-Reset Discharge
F	Fuse SA
PL	Pilot Green
K-1	Mercury 3PST, 2NO, Ebert, INC EM-7
K-2	Mercury DPST, 2NO, Ebert, INC MR-14
SR	Microswitch (Recorder)
R-1	Ampere Shunt, 10 Ohm 10 Watts Low Range
R-2	Load Resistance
R-3	Load Resistance, 2 Ohm, 10 Watts High Range

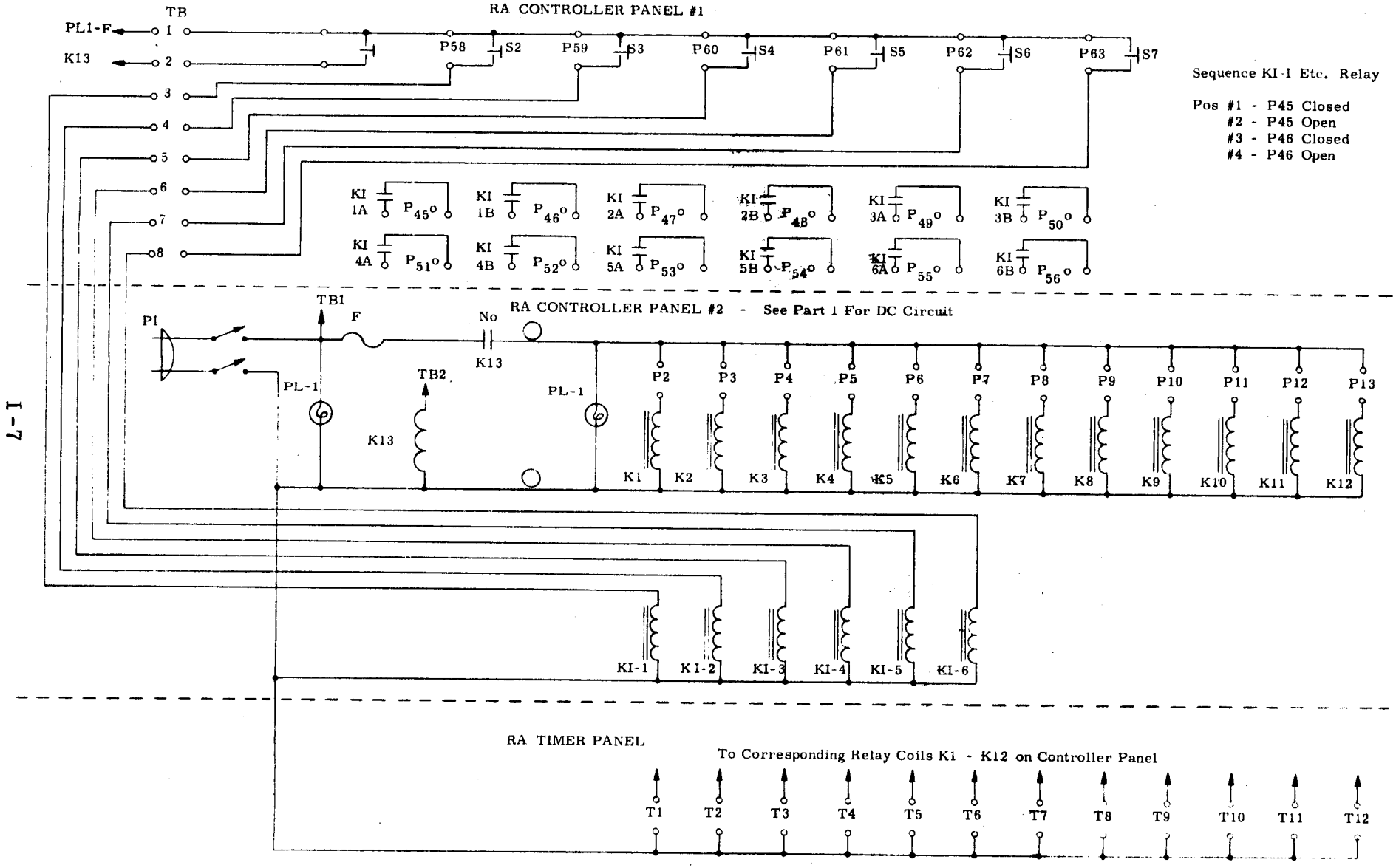
AC Circuit on PT2 Sketch

RA Controller Panel Center Section

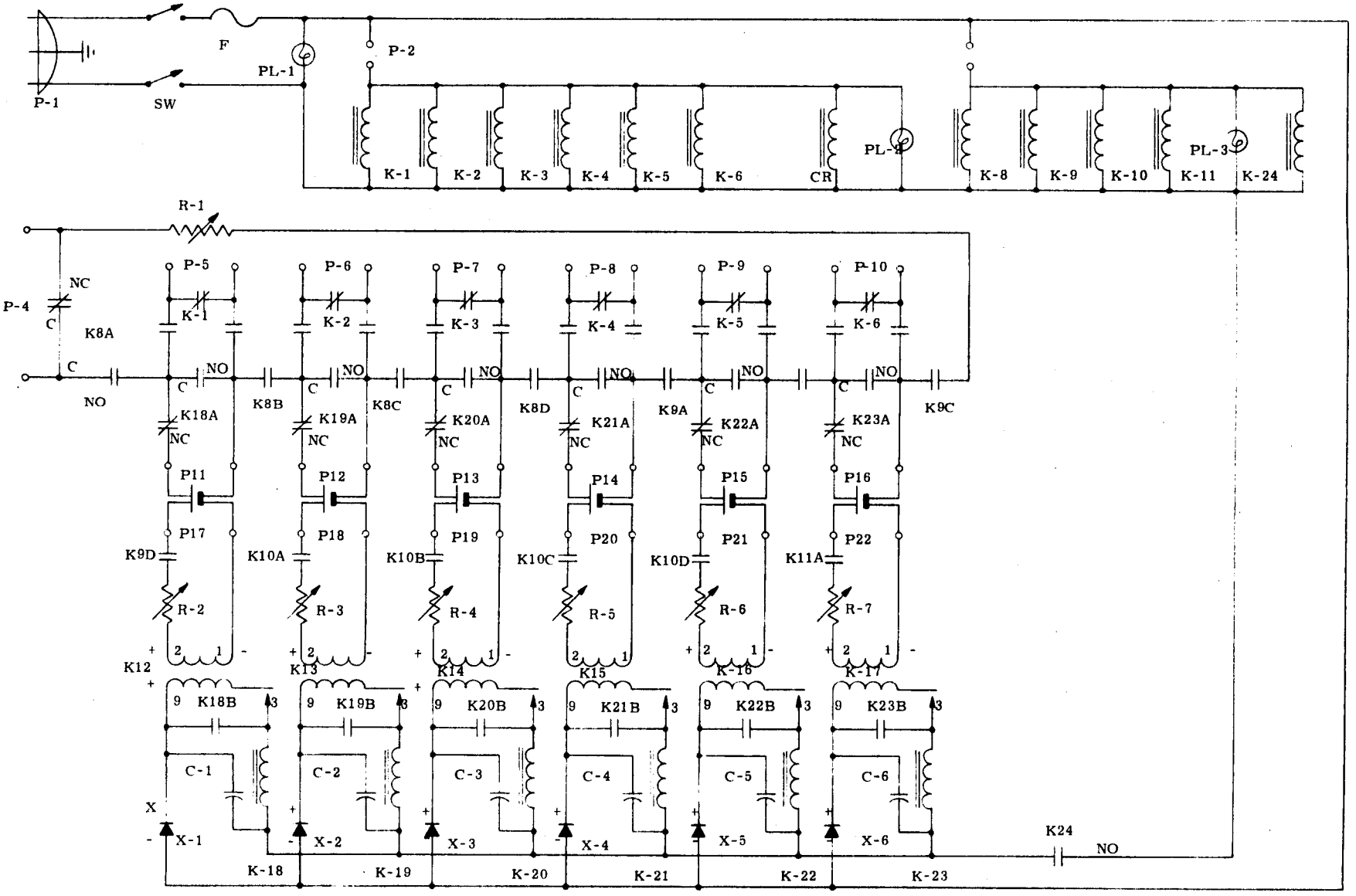


I-6

Ni-Cd Characterization
RA Controller
Part 1 of 3 Parts



Ni-Cd Characterization - RA Controller - Pt. 2 of 3 Pts.


P-1
SW
F
PL-1
P-2
K-1
K-2
K-3
K-4
K-5
K-6
CR
PL-2
K-8
K-9
K-10
K-11
K-24
PL-3
R-1
P-4
NC
C
K8A
NO
P-5
P-6
P-7
P-8
P-9
P-10
K18A
K19A
K20A
K21A
K22A
K23A
K8B
K8C
K8D
K9A
K9C
P11
P12
P13
P14
P15
P16
P17
P18
P19
P20
P21
P22
K9D
K10A
K10B
K10C
K10D
K11A
R-2
R-3
R-4
R-5
R-6
R-7
K12
K13
K14
K15
K16
K17
K18B
K19B
K20B
K21B
K22B
K23B
C-1
C-2
C-3
C-4
C-5
C-6
X-1
X-2
X-3
X-4
X-5
X-6
K-18
K-19
K-20
K-21
K-22
K-23
K24

PARTS LIST
RA CONTROLLER

Part 3 of 3 Parts

P-1 Plug, Line Cord Male, 110 Volt Input

P-2, P-4, P-6, P-8, P-10, P-12 Charge Inputs, Yellow Jacks

P-3, P-5, P-7, P-9, P-11, P-13, Discharge Inputs, Red Jacks

P-14 Charge Power, Yellow Jacks

P-15 Discharge Power, Red Jacks

P-16 thru P-39 Cell Inputs, Red + Black -

P-45, P-47, P-49, P-51, P-53, P-55 Charge Outputs, Yellow Jacks

P-46, P-48, P-50, P-52, P-54, P-56 Discharge Outputs, Red Jacks

P-57 Lamp Input, Line Cord

S-1 Master Switch, DPST Toggle

S-2 thru S-7 Push Button, No Relay Synchronization

PL-1 Pilot, Master, Green

PL-2 Pilot, Relay Power Amber

F Fuse, 5 Amp.

T-1 thru T-12 Timer, Elapsed Time, Cramed 640E Reset Meter, 115 VAC Coil, 9999. 9 Minutes

K-1 thru K-12 Relays, DPOT, 115 AC Coil Leach 337

K-13 Lamp Relay (Safety) 12UAC Coil Ebert MR10-SPST-No

KI-1 thru KI6 Ratchet Relay Guardian 1R-M-120-115, DPDT 3 Positions Sequence Ratchet

R-10 Resistor, Load (25 Ohms, 12. 5 Watts, Ohmite 0113

PARTS LIST
RB CONTROLLER

Part 2 of 2 Parts

P-1 Line Cord 110V AC Power Input

P-2 Control, Charge Pin Jacks, Yellow

P-3 Control, Discharge Pin Jacks, Red

P-4 Input Terminal, Discharge CCS Terminals

P-5 thru P-10 Input Terminal Charge CCS'S Terminals

P-11 thru P-16 Cell Input, Power, Pin Jacks; Red +, Black -

P-17 thru P-22 Cell Input, Control, Pin Jacks, Red +, Black -

SW Switch DPST Toggle Master

F Fuse 3A

PL-1 Pilot, Green, Power On

PL-2 Pilot, Yellow, Charge On

PL-3 Pilot, Red, Discharge On

CR Counter General Controls CE 600B5602

K-1 thru K-6 Mercury Relay 3PST 2NO, INC Ebert EM7 KOVAC Coil

K-7 Deleted

K-8 thru K-10 Leach 329-7 4PDT 110 VAC Coil

K-11 Leach 337 2PDT 110 VAC Coil

K-12 thru K-17 VHS Meter Relay, Low Limit Contacts, 100 Microamperes 137 (Assembly Prod).

K-18 thru K-23 P&B LM 11, 5000V DPDT

K-24 GE - Time Delay Switch - 1 min. - No

R-1 Load Resistance, 6 Ohm, 25 Watts, Ohmite O143

R-2 thru R-7 10,000 Ω Pot WW

C-1 thru C-6 Condenser SMFD, 200 Volt

X-1 thru X-6 Rectifier